From:	DeVisser, Anita
To:	Bradley, John (DEQ)
Cc:	Fortenberry, Chase; Griffith, Garry T.; Draper, Cynthia E; Bondy, Garret E; Abid, Joseph A; Saric, James; Santini, Andrew; Jeff.Keiser@CH2M.com; Bucholtz, Paul (DEQ)
Subject:	FW: Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments
Date:	Thursday, July 11, 2013 7:42:35 AM
Attachments:	BR-GT-021r1_Grind & MIS.pdf

Hi John – please see the response from TestAmerica and the attached (updated) SOP that the Burlington Lab uses. The Burlington lab will be processing the incremental samples. Anita

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From: Madison, Jim [mailto:Jim.Madison@testamericainc.com]
Sent: Wednesday, July 10, 2013 2:06 PM
To: DeVisser, Anita
Subject: RE: Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments

Hi Anita.

I'm attaching Burlington's grind SOP. We do not have as many ISM options as Canton, but routinely air dry, break up agglomerates and sieve through a #10 sieve (without using the puck mill). We then do the 2-D slabcacke subsampling. We have historically done automated soxtherm extraction for kalamazoo PCBs, which uses 15 grams of sample. We could sonnicate instead, using 30 grams if necessary.

Jim.

From: DeVisser, Anita [mailto:Anita.DeVisser@amec.com]
Sent: Wednesday, July 10, 2013 1:40 PM
To: Madison, Jim
Subject: FW: Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments

Hi Jim
Here is a bit more information for the IS sample prep. Are you available this afternoon for a call?

From: Bradley, John (DEQ) [mailto:BRADLEYJ1@michigan.gov]
Sent: Wednesday, July 10, 2013 1:35 PM
To: DeVisser, Anita
Cc: Draper, Cynthia E; Bondy, Garret E; Fortenberry, Chase; Griffith, Garry T.; Abid, Joseph A; Bucholtz, Paul (DEQ); saric.james@epa.gov; 'Jeff.Keiser@CH2M.com'; King, Todd W.; Santini, Andrew
Subject: RE: Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments

Anita

Thank you for the summary and the attached SOP

In regard to comment #5 below…

I have attached the more recent TestAmerica Subsampling SOP containing Incremental Sampling (IS

or ISM) procedures.

The SOPs provide multiple procedures for various objectives. The specific procedures that we (and the lab) plan to use on this project need to be identified in consultation with Georgia Pacific, AMEC, and TestAmerica. The following come to mind.

- Section 11.3.2 identifies different methods for mixing and particle size reduction. There are no milling techniques mentioned other than hand milling by mortar and pestle of the coarse fraction. Mortar and pestle of the coarse fraction should be sufficient.

- Section 11.3.2.4.1. "Air dry, chop, sieve, mix" is essentially dry, disaggregate, and sieve. A number 10 sieve (2mm) should be appropriate.

- Section 11.3.3.2 “Subaliquoting Procedures” include a number of procedures. Two-dimensional slabcake is the appropriate “Solid Sample Subaliquoting Procedure” to be used for our purposes.

- Procedures for subsample (aliquot) preparation for extraction and “method of extraction” need to be specified. TestAmerica typically uses 30 g for PCB soil prep for Soxhlet (Method 3540) and sonication (Method 3550) extraction and 5 g for microwave extraction. The larger subsample is normally used for these purposes and will help manage heterogeneity still present in the sample after the IS processing. The 30g Soxhlet prep is recommended.

Please share this with your TestAmerica contact so he can provide his perspective/confirmation. Perhaps he has the SOP for Soxhlet prep and extraction as well.

Thanks

John

From: DeVisser, Anita [mailto:Anita.DeVisser@amec.com]
Sent: Tuesday, July 09, 2013 1:54 PM
To: Bucholtz, Paul (DEQ); Bradley, John (DEQ); saric.james@epa.gov; 'Jeff.Keiser@CH2M.com'; King, Todd W.; Santini, Andrew; roth.charles@epa.gov; Canar.john@Epa.gov
Cc: Draper, Cynthia E; Bondy, Garret E; Fortenberry, Chase; Griffith, Garry T.; Abid, Joseph A; DeVisser, Anita
Subject: Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments

Provided below are responses to comments received via email from the MDEQ on June 27, 2013 for the Area 3 Field Sampling Plan. The responses are representative of our phone conference held July 2, 2013, discussion of the issues, agreements and conclusions. In addition, attached is the TestAmerica SOP for processing the incremental samples.

Incremental Sampling

1. One procedure had been discussed was conducting limited hand augers in the area of the incremental sampling to confirm the underlying stratigraphy. If we did not overlook the detail in the plan, MDEQ believes it is prudent to conduct some limited hand auguring in the areas of the incremental sampling to confirm the underlying stratigraphy of the sample material. Response: Bank sampling in the residential area will be conducted prior to the incremental sampling in the backyards. This approach will help AMEC to identify the "interval of interest" before conducting sampling in the backyards and give information on underlying stratigraphy that may be encountered in the backyards. AMEC recommends that any additional coring in the yards be conducted nearer to the river and not in every yard – especially not the well manicured lawns. MDEQ agrees that we should minimize augering in the yards, and if it becomes necessary to confirm the interval of interest, the boring would be conducted nearer to the river rather than closer to the house.

2. MDEQ notes that the residential areas may be areas of high heterogeneity. This is expected due to the distributional heterogeneity typical of floodplains and because the DUs proposed include both exposed (floodplain) and unexposed (upland backyard) "populations". The use of 30 increments is considered minimal and may result in higher than decided error, dependent upon our goals. It may be prudent to increase the number of increments to 49 (7 x 7 grid) to handle the anticipated heterogeneity. At this point it is not clear what number of increments is a best fit and will depend on heterogeneity of the area, sampling goals, and the proximity of the sample results to selected criteria. AMEC agrees that precision error would be reduced by increasing the number of increments. Based on the general rectangular shapes of the backyards, AMEC suggests a different grid shape (8 x 6). MDEQ agreed with this approach.

3. From a process standpoint we think it will be prudent to identify the sampling procedures before we get in the field. For example:
    a. Will both intervals be collected from one push of the probe or will intervals be collected separately.
    a. If collected together and full target depth of 12 inches is not recovered, identify how the aliquots are to be split into the 0-6' interval and 6-12' interval)
    b. The specific tool to be used is important (in order to predict the proper amount of sample mass).
    c. Identify a recovery minimum at which point resampling will be conducted.

AMEC tested the equipment, a soil push-probe (7/8" diameter) during site recon to evaluate the collection of the two intervals and recovery. The push probe appears to be appropriate for collecting both intervals with good recovery. MDEQ is pleased with our equipment and field experience. The target is 75% recovery. Both AMEC and MDEQ acknowledge that site conditions may vary across the residential yards, and additional equipment (such as a spade-shovel) may be needed to collect samples at the predetermined intervals. MDEQ was concerned with potential cross contamination between 0-6" and 6-12". AMEC to follow up with research. AMEC and MDEQ agreed that if it appeared that there were apparent smearing (of gray material) between the two intervals, 2 push probe samplers would be a good solution, one to 6" and the other 6-12".

4. It is not clear how the eco assessment will be conducted. It appears that it will be conducted over an approximately 2000 foot stretch of the bank as one DU. Please clarify if multiple DUs will be sampled along the bank. Also, it is not clear if the 683 NGVD will be used as a hard line for eco sampling, or if the exact extent of the DU(s) will be based on field observations as well.

AMEC intends to establish the 683 elevation and use the area from this elevation to the water as the DU. However, given our observations during site recon, and topographic map information, there may not be much distance between the 683 elevation and the water, if any. As a backup plan, AMEC would mark off a strip 20 foot wide along the river, across contiguous residential properties. MDEQ agrees with this approach and backup plan. AMEC explained that goal is contiguous, but may be broken due to access issues. AMEC agreed to do triplicate in the ecological DU.

5. A critical component of IS is the laboratory processing of the IS sample after it is collected in the field. The specific lab and the specific lab processing procedures (including the specific subsampling, aliquot mass and analytical methods) will be important to identify before the sampling event.

AMEC plans to use TestAmerica Lab (Canton, Ohio) and their existing SOP. Disaggregation of the sample through crushing, and not grinding will prevent smearing. MDEQ agrees with the method. AMEC's contact at the lab is Mark Loeb. AMEC to provide laboratory SOP to MDEQ.

Bank Sampling

6. It appears from the FSP that different sample core processing is being proposed. MDEQ believes that retaining the previously approved processing regimen is best. For example, collecting cores with 3" Lexan tubes in lifts as appropriate, and dividing samples on the 0-6, 6-12, 12-24, etc. with segregation of material of interest. We understand that the floodplain in area 3 may require hand augering through the hard upper material, before proceeding with Lexan.

AMEC agrees with MDEQ intervals (0-6, 6-12 and 12-24) to be consistent with past sampling. Anita noted that the interval 12 to 24" may be saturated. MDEQ recommends sampling even if saturated. AMEC agreed to collect even if saturated. AMEC asked about specification of 3" vs. 2" diameter Lexan tubes. MDEQ recommends using the 3" diameter Lexan tubes due to their experience with less compression during advancement and better recovery. AMEC agreed to use the 3" Lexan tubes.

Mill race

7. The inclusion of 4 transects from the river edge, further upstream the former race to the parking lot area is appropriate. AMEC agreed to add another transect "upstream" in the old mill race and then evenly distribute the transects.
8. Make sure transects only occupy former race, as the line (in blue) depicts the race as being wider than aerials suggest. The width of the transects illustrated on Figure 4 may lie outside of the mill race channel. The MDEQ suggested that the transects be located in the field based on observations of topography and avoidance of bank soils. AMEC agrees with this method of transect placement.
9. Once all cores are collected along the transect, the Lexan should be observed and the most interesting core sent for analysis (as opposed to only selecting the middle core). Other cores sent to freezer pending results. Previous sampling results from the mill race area had highest concentrations of PCBs at the 681-682 elevation; AMEC believes it is important to reach this depth with advancement of hand auger and/or Lexan tubes. AMEC suggested that all samples collected along the transects be analyzed to provide more complete

characterization of extent. The MDEQ agrees with this approach.

10. For all cores use traditional sectioning. Although AMEC had initially targeted the 681-682 elevation for sample collection, the method proposed by MDEQ (and previously employed) to collect samples at the 0-6", 6-12", 12-24" intervals and additional samples at intervals of interest within these predetermined intervals is an acceptable alternative. MDEQ noted that there were some sampling guidelines developed by the MDEQ to facilitate this type of sampling and they will provide these guidelines to AMEC.

Anita Emery-DeVisser
Project Manager/Senior Scientist

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Title: Sample Grinding & *MULTI INCREMENT*® Sampling Procedure (SW-846 8330B)

Approval Signatures:

William S. Cicero
Laboratory Director

Chris G. Callahan
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Bryce E. Stearns
Technical Director

Dan Helfrich
Health & Safety Coordinator

Approval Date: May 3, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

SOP REVIEW FORM

SOP Number	Revision	Effective Date:	Title
BR-GT-021	1	05/03/10	Sample Gravity + MS Procedure

Review Statement:

My signature signifies that I reviewed and compared the above referenced SOP against current bench practice.

			Revision Needed	
Date	Reviewer	Job Title	Yes[1]	No
7/31/12	Christopher Callahan	Dept Manager		X

[1]: *List the section for revision and the the reason using the revision summary page and attach to this cover sheet.*

QA Use Only:

✓ The SOP was reviewed and does not require revision. Attach this form to the SOP.

_____ The SOP Revision will be made with a Change in Progress Attachment (CIPA).

_____ The SOP Revision will be released as a new version of the SOP.

_____ The SOP Revision requires method validation or demonstration of capability

_____ The SOP Revision does not reqire method validation or demonatration of capability.

_____ The SOP revison affects other SOPs that must now also be revised (List SOPs)

_____

_____

Kristi Daigle
QA Signature

07/31/12
Date

Comments:

1.0 Scope and Application

This SOP describes the laboratory procedures for the preparation of soil and sediment samples collected for *MULTI INCREMENT*® Sampling (MIS) for SW-846 Method 8330B.

This SOP may be modified and applied to samples for other determinative test methods on a project basis. Project specific procedures are determined jointly between the laboratory and the client and are documented in the project record.

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP may be used for a variety of matrices including: Soil, sediment and sludge.

This SOP is a preparatory procedure. Refer to the test method SOP to review target analyte lists and reporting limits.

2.0 Summary of Method

The entire sample mass, usually one kilogram dry weight, is air dried and passed through a #10 sieve to exclude material greater than 2 mm in size. For energetic constituents, the material finer than 2 mm is ground for 90 seconds in a mechanical mill in order to reduce the particle size to less than 75 microns. For samples containing nitrocellulose based propellant residues, the sample is ground for 5 minutes in a mechanical mill in order to adequately pulverize the sample. After the grind, the sample is spread on a clean surface and at least 30 sample increments are randomly selected to create one or more sub-samples.

This procedure is based the following reference documents:

- SW-846 Method 8330B, Appendix A, Revision 2, October 2006.
- Guide for Implementing EPA SW-846 Method 8330B, DoD Environmental Data Quality Workgroup, June 2008.

If the laboratory's procedure is modified from the reference documents, a list of method modifications will be provided in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

The MIS procedure outlined in the reference method stipulates that the entire sample collected from the field must be ground. Due to potential contamination or the loss of heat-sensitive compounds during grinding, it may not be advisable to grind the entire sample if the subsample is to be used for analyte groups other than explosives, such as metals or semivolatile organics. Considerations to subsample a portion of sample prior to grinding and MIS should be made on a project basis with specific client instructions provided to the laboratory.

The grinding procedure produces a very fine material which can easily become airborne and care must be taken to prevent cross-contamination. Grinding bowls should always be opened in a

low-flow fume hood, which must be decontaminated between samples.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Protective clothing such as a lab coat, safety glasses and latex or nitrile gloves must be worn when performing this procedure.

The grinding procedure involves the use of metal bowls weighing up to 45 pounds. Analysts should be capable of lifting the metals bowls from one work station to another without fear of dropping them on their feet or pinching their fingers. If any analyst is not comfortable lifting these metal bowls, the analyst must notify their department supervisor and request re-assignment of duties.

This procedure is for the preparation of solid samples that may contain energetic residues less than 2 mm in size, a particle size that can be safely processed. Samples that have energetic materials larger than 2 mm will not be accepted by the laboratory. In general, secondary explosives in excess of 12% purity can detonate when processed using this procedure. Although concentrations at this level of purity are rarely encountered and contractors submitting samples to the laboratory have procedures designed to screen the samples for pure energetic materials, all samples must be visually inspected in the laboratory. Samples that contain lumps of material that have a chemical appearance (explosives are generally a grayish-white or reddish-brown in color) should be set aside and the laboratory project manager (PM) immediately notified who will subsequently notify the client to determine the next course of action.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

- Puck Mill, LabTech Essa LM-2 Ring Mill or equivalent.
- Bowl & Puck, 800 cc, Lab Tech. 3 sets recommended for production purposes.
- Optional Bowl & Puck, 1000 cc Lab Tech.
- Commercial Baking Tray, 18 x 30”
- Drying Rack, equipped with a cover to minimize ultraviolet light exposure. The drying rack should be connected to external exhaust to create gentle air flow.

- Aluminum Foil, Heavy-duty 18 x 30”
- Stackable Sieve, 12”, #10 (2.00 mm), Gilson Company, Inc. or equivalent.
- Stackable Sieve collection pan, 12”, Gilson Company, Inc. or equivalent.
- Sieve, #4 (4.75 mm), Gilson Company, Inc. or equivalent
- Sieve Shaker, Humboldt or equivalent
- Grinding media, Fisher or equivalent
- Stainless steel spatulas and spoons, Fisher or equivalent.
- Polypropylene Sample Bags, NASA JPG 5322.1 Level 100 or equivalent (seamless, round bags 15 x 20”, 6 milliliters thick, that fit around a sieve).
- Top Loading Balance capable of measuring 20 Kg +/- 1 gram.
- Top Loading Balance capable of measuring 4000 grams +/- 1 gram.
- Drying rack which holds disposable polypropylene pipet tips to facilitate drying of sample bags.
- Disposable poly pipet tips, Thermo/Fischer Finntip 5ml or equivalent.
- Plastic Bags, Quart size, Zip-Loc or equivalent.
- Optional: Sieve, Teflon lined, #10 (2.00 mm), Gilson Company, Inc. or equivalent.
- Optional: Heavy-duty parchment paper
- IR Gun, 50.0 to 1000 C, Fisher Scientific or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Acetone: Pesticide Quality, J. T. Baker or equivalent.

7.2 Standards

- Solid Reference Material (SRM): Custom Standard #093 USACE Explosives in Soil, Environmental Resource Associates or equivalent.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Listed below are minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation[1]	Holding Time[2]	Reference
Solid	Polyproplylene Bag	1 Kg (wet)	Chilled to 4°C	14 days	SW-846 8330B

[1]*Sample is maintained under thermal preservation until time sample is air dried, after which, ground samples are held at room temperature protected from light.*

[2]*Holding time is determined from date of sample collection.*

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

The laboratory prepares the following quality control samples with each grind batch of up to 20 samples.

QC Item	Frequency	Acceptance Criteria
Grinding Blank (GB)	After every sample	See Test Method SOP
Solid Reference Material (SRM)	1 per grind batch of 20 or fewer samples	See Test Method SOP
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1 per grind batch of 20 or fewer samples	See Test Method SOP
Sample Triplicate (ST)	1 per grind batch of 20 or fewer samples per customer request.	See Test Method SOP

9.1 Equipment Verification

The laboratory must perform a one-time demonstration to show that the grind mill is able to reduce particle size to less than 75 um. To verify equipment capability, analyze a sample that was subject to this procedure by ASTM D4464 Particle Size Laser Light Analysis using laboratory SOP BR-GT-005.

10.0 Procedure

10.1 Equipment Cleaning

After each use, scrub all surfaces of the bowl, puck and lid with hot soapy water. Rinse with tap water. Let the puck and bowl sit full of cold tap water in order to reduce the temperature before they are used again. Dry the bowl, puck and lid with clean paper towel then rinse lightly with acetone to remove any residual moisture. Use care to remove any moisture trapped underneath the red O-ring on the lid, and in the recessed notch of the puck.

The low-flow hood must be vacuumed, washed down with soapy water, rinsed with reagent water and towel dried prior to each use in order to remove all dust that may have been generated by the processing of the previous sample.

10.2 Sample Drying

Line a clean baking tray with heavy-duty aluminum foil. Label the edge of the foil with the sample's laboratory ID (LAB ID). Place the foil-lined tray on a top-loading balance and record the tare weight on the bench sheet. Remove the tray from the balance.

Ensure the sample container is labeled with the sample's LAB ID. Open the sample container and empty the sample into tray. Using your glove-protected hands manually spread the sample evenly across the tray. Break up any clumps to speed up the drying process and to ease sieving. Re-weigh the tray + sample and record this weight as the "as received" sample weight on the bench sheet.

Place the empty sample container on a drying rack and allow to air dry. Place the baking tray + sample on the drying rack and dry for 24-48 hours. If a sample is predominantly wet, break-up of agglomerates after 8-24 hours of drying time. Record the date and time samples were set to dry

on the bench sheet. Record the room temperature on the bench sheet. Measure and record the temperature each day of drying time.

Visually inspect the sample and when the sample appears dry, re-weigh the sample. The sample is considered dry when consecutive measurements agree within 1% tolerance. The recommended wait time between consecutive measurements is 1 hour.

If any residual sample material dried to the surface of the sample's original container, transfer the residual sample material to the rest of the sample on the aluminum foil then re-weigh to determine the post-dry weight of the sample.

10.3 Sample Seiving

Clean and rinse each stackable sieve and collection pan with acetone prior to use. Label each stackable sieve and collection pan with the corresponding sample ID. Place a #10 sieve on top of a collection pan this setup is referred to as a sieve set.

Add the sample and 25-30 pieces of grinding media to the sieve set. Repeat the previous steps for a new sample using a clean labeled sieve set 2 more times, so there are 3 separate samples and sieve sets.

Load and fasten the stackable sieve sets onto the sieve shaker. The sieve shaker can accommodate three sieve sets at a time. After the sieve sets are securely fastened, set the timer on the sieve shaker for 15 minutes close and lock the sound enclosure. After 15 minutes remove sieve the sets from the shaker and apply light pressure as needed with a gloved hand or spoon to pass the sample through the #10 sieve and into the collection pan. Break up soil agglomerates that formed during drying but do not force actual particles greater than 2mm material through the sieve. If necessary, sieve the air dried sample through a #4 sieve to remove larger stones.

NOTE: Particulates of fine vegetation may pass the #10 sieve. This material may entrap crystalline or fibrous energetic residues, so this material should not be removed from the sample unless the samples were collected at a time long past the time period when the site was actively used as a firing range. In these instances, any vegetation present is not related to the vegetation that grew when the firing range was active. Per client instructions, such vegetation may be removed on a project basis. If removal of vegetation is required, the laboratory PM must provide such instruction to the laboratory operation using the comment section of the LIMS system or a project plan. The default procedure of the laboratory is to include all material that passed the #10 sieve.

Transfer the retained material from the top of the #10 sieve to a tared, 1 quart re-sealable plastic bag, weigh and record this mass on the bench sheet. Record the presence of any foreign materials (metal, shrapnel, bullets, etc.) on the bench sheet.

10.4 Sample Grinding

NOTE: Samples to be analyzed for metals or mercury should not be ground. If the sample is to be analyzed for these analyte groups, after the sieving step, proceed to Section 10.5 to prepare the MIS subsample. After the subsample is taken, return to this section to grind the sample.

Determine the number and mass of aliquots needed for each sample. The maximum mass of sample that can be ground at one time in the 800 cc bowl is 600 grams. To determine the number of aliquots required per sample, divide the total post-dry sample mass by 600 and round up to the next whole number. To determine the mass of sample per aliquot, divide the total post-dry sample mass by the number of aliquots required. For example, if the total mass of sample is

950 g, the sample will require 2 aliquots of 475 g each. The length of time that each aliquot must be ground is specified by the project.

Transfer the appropriate mass of sample to the bowl. Slowly place the puck on top of the sample, so that it touches the side of the bowl and place the lid on the bowl. Transfer the bowl to the grinding mill and set the mill for the proper grind time as specified in the project notes.

Start the mill and listen to be sure it achieves a balanced hum, which indicates that the puck is smoothly rotating inside the bowl. The hum should be heard within ~30 seconds from the start of grind time. If the puck fails to rotate properly, shut down the mill, remove the bowl assembly, reposition the puck in the bowl and retry.

Using the IR gun, monitor the temperature of the puck after each grinding to ensure it has not risen above 30°C. In the temperature exceeds 30°C, immediately notify the PM, who must subsequently notify the client. If the temperature is approaching this threshold, cool the bowl to ambient or less before using it to grind additional sample aliquots . In general, 90 second grinds do not generate sufficient heat to necessitate using multiple bowls for up to three aliquots. The 5 minute grinds typically reach temperatures of up to 27°C and for this reason the laboratory uses multiple bowl sets for larger samples (greater than 600 g post-dry mass).

Line a clean low-flow fume hood with heavy duty aluminum foil. After grind is complete, move the bowl from the mill to the fume hood. Gently remove the lid and set it down on a corner of the foil. Gently remove the puck and set it on another corner of the foil. Slowly invert the bowl to slide the ground sample to the foil. Gently tap the bowl as it is inverted to ensure a complete transfer.

Note: Use caution when transferring the ground sample to the foil. The grain size of the sample is less than 75 microns and resembles a fine powder. If the sample dumps out suddenly, it will become airborne and some sample material may be lost on the inside of the fume hood.

Continue the grind step for each sample aliquot needed to grind the entire sample. If temperature monitoring of the puck suggests that the temperature is nearing the maximum threshold, use a separate bowl and puck for each additional aliquot (or allow the bowl set to cool before continuing). Clean the bowl, puck and lid (grind equipment) after each sample, performing a grinding blank (See 10.4.1) each time the grind equipment is used.

After the entire portion of sample is ground proceed to Section 10.5.

10.4.1 Grinding Blanks

A grinding blank must be performed after each bowl set (bowl, puck and lid) for each sample is cleaned to verify cleanliness. To prepare the grind blank (GB), transfer 300 g of Ottawa Sand to the grind bowl and grind the blank using the same procedure described for samples in Section 10.4. After the grind is complete, transfer the GB to a 1 quart re-sealable plastic bag and label the GB with “GB” followed by the sample’s LAB ID using a sequential alpha character to differentiate between multiple grind blanks associated with a single sample. For instance, using the same example provided earlier, a sample (Lab ID 12345) with a total post-dry sample mass of 950 g and a grind time of 5 minutes will require two 475 g aliquots ground in two different bowl sets. The GBs for this sample would be labeled “GB12345A” and “GB12345B”, one for each bowl set.

After all samples in a grind batch are complete, a composite of all of the grind blanks is prepared, this procedure is described in Section 10.5.

10.4.2 Standard Reference Material (SRM)

Grind one SRM with each grind batch. After allowing the SRM material to equilibrate to room

temperature, transfer 300 g the SRM into a clean, tared grinding bowl and record the mass on the bench sheet. Lay out a clean sheet of aluminum foil in the low-flow hood and transfer the bowl to the foil. Carefully place the puck on top of the SRM sample, making sure that the edge of the puck is in contact with the bowl. Cover the bowl with the lid, place the bowl set in the mill and follow the grinding time requirements used for all of the samples in the batch. After grinding, return the bowl set to the foil, uncover and carefully remove the puck. Slowly transfer the ground SRM sample to the foil. Spread the sample out on the foil to create a uniform depth of ¼ - ½ inch. Collect 10 g using at least 30 random aliquots of ~0.3g into a tared, labeled 40 mL amber vial. Transfer the remaining ground SRM sample to a 1 quart re-sealable plastic bag, label with batch information and store with the rest of the samples in a dark, room temperature location.

10.5 Multi-Increment Sampling (MIS)

When the grind for an entire sample is complete, mix the sample using a spoon then spread the sample evenly across the foil to approximately ¼ to ½ inch thick.

Label a 40 mL vial with the sample's LAB ID. Place the vial on a top-loading balance and tare the vial. Take at least 30 random aliquots from the sample and transferring each aliquot to the vial. Each aliquot must include the entire depth of the sample and the combined weight of the 30 aliquots should approximate 10 g. If the weight of aliquots taken is not at least 10 g, continue to add additional aliquots until the desired weight is achieved. If the weight of aliquots taken exceeds 10g, dump the vial and start over or thoroughly mix the contents of the vial and remove small portions of sample, mixing between each portion until the 10 g weight is achieved. Record the final weight on the bench sheet. Transfer any remaining sample to its original container and place the container in storage.

Prepare separate MIS samples for the designated matrix spike and matrix spike duplicate (MS/MSD). If no sample is specified as the MS/MSD randomly select a sample from the grind batch to serve as the MS/MSD.

Select one sample from each grind batch to serve as the sample triplicate (ST). Prepare two separate MIS samples for the sample triplicate. Append an A1 suffix to the LAB ID of one vial, and an A2 suffix to the LAB ID of the other.

Prepare an MIS sample for the SRM using the same procedure described for samples.

To prepare the composite grind blank (GBC), weigh 10 g aliquots of each individual grind blank from the grind batch into a new, clean 1 quart re-sealable plastic bag. Lay a clean sheet of foil down, dump the GBC onto the foil and subsample at random 30 aliquots using the same technique as used for samples.

Return the remaining portion of the individual grind blanks to storage.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

This section is not applicable to this procedure.

11.2 Quantitative Identification

This section is not applicable to this procedure.

11.3 Calculations

This section is not applicable to this procedure.

11.4 Data Review

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Retain, manage and archive electronic and hardcopy data as specified in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

This section is not applicable to this procedure.

12.2 Demonstration of Capabilities (DOC)

See Section 9.1 for equipment verification.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Solvent Waste -Satellite Container: 55 Gallon Steel Waste Drum

Transfer the waste stream to the appropriate satellite container(s) located in your work area. Notify authorized personnel when it is time to transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations. The procedures for waste management are provided in the laboratory SOP for hazardous waste,

15.0 References / Cross-References

- SW-846 Method 8330B, Appendix A, Revision 2, October 2006.
- Guide for Implementing EPA SW-846 Method 8330B, DoD Environmental Data Quality Workgroup, June 2008.
- Corporate Environmental Health and Safety Manual (CW-E-M-001), Current Revision.
- Laboratory SOP BR-EH-001, Current Revision.
- Laboratory SOP BR, GT-005, Current Revision
- Laboratory SOP BR-QA-014, Current Revision.
- Laboratory SOP BR-QA-002, Current Revision.
- Appendix A: Terms & Definitions

16.0 Method Modifications

Modification Number	Method Reference	Laboratory Modification
1	EPA Method 8330B	The laboratory performs one 5 minute grind of nitrocellulose samples instead of 5 one minute grinds.
2	EPA Method 8330B	Soil Sample Triplicate are performed per customer request.

17.0 Revision History

BR-GT-021, Revision 0:

- Initial Release of SOP

BR-GT-021, Revision1:

- Title Page: Updated Approval Signatures
- Section 1: Addition of reference to Appendix B for method modifications of the MMR site.
- Section 6.0: Changed sieve size from 8” to 12”
- Section 6.0: Addition of sieve shaker and grinding media
- Section 10.3: Addition of procedure for mechanical sieve shaker using stackable sieves.

18.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions
- Appendix B: MMR Site Modification Procedure for Grinding and MI sampling 8330B Solids

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness and headache.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Appendix A: Terms and Definitions

Agglomerate: A jumbled mass of solid material composed of fragments of various sizes and angularity. In the application of this procedure, agglomerates are most commonly created when wet samples containing some clay are dried. The resulting clumps of material are agglomerates.

Batch: Environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Energetics: Material residues of unreacted explosives and propellant compounds that remain after firing or the detonation of munitions.

Holding Time: The maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Matrix Spike (MS): A field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): A duplicate field sample to which a known amount of target analyte(s) is added. Used to assess precision.

Multi Increment Subsample: A sample collected from a minimum of 30 points spread over a decision unit. For clients, an MIS decision unit may be an entire field. For the laboratory, the MIS decision unit is the entire sample submitted to the lab, spread out after processing.

Non-confirmance: An indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: Refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Puck Mill: A mechanical grinder capable of reducing solid samples to a grain size of 75 um or less. An example is the LabTech Essa LM-2 Ring Mill. The mill is outfitted with bowls which contain the sample and pucks which rotate inside the bowl mechanically reducing the grain size of the sample.

SRM: Solid Reference Material. A sample with a known concentration of the target constituents. Currently, Environmental Resource Associates (ERA) offers Custom Standard No. 093, labeled USACE Explosives in Soil.

Appendix B: Common Modifications for Grinding and MI Sampling for SW-846 8330B.

This appendix describes a site specific project specific procedure used to prepare samples for 8330B. This procedure may not be used for other project work unless the procedure modifications are stipulated in writing by the customer for each project.

Follow all procedures described in the main body of the SOP except:

1. Perform a grind blank every 10 or fewer samples instead of after every sample and do not composite the grind blanks. For example, a batch of 20 samples will include 2 grind blanks. These grind blanks are not composited but subsequently extracted and analyzed as separate samples.

2. Analyze the SRM at the frequency specified by the customer instead of with each batch. On an annual basis, perform a test that includes a method blank and 4 SRM replicates taken through the grind and MIS procedure and subsequent extraction and analytical procedure for 8330B.